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Determination of particulate carbon-water quality
section report number 3.

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DETERMINATION OF
PARTICULATE CARBON

WATER QUALITY SECTION
REPORT NUMBER 3

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by:
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INTRODUCTION

The objective of the carbon project is to develop methodologies for the following parameters in aqueous media: volatile organic carbon, dissolved organic carbon, particulate organic carbon, carbon dioxide, dissolved inorganic carbon, and particulate inorganic carbon. Throughout this project it is presumed that volatile organic constituents are not present in particulate matter. Reports on dissolved organic and inorganic carbon have been issued^{1,2} as well as a preliminary report on the use of the Leco WR12 system³. The following report describes a procedure for the determination of total carbon in particulates using the Leco CR-12 carbon system.

The Leco CR-12, a new instrument produced for the environmental market, is designed primarily to determine the percentage carbon in a solid sample. The self-contained unit consists of a 3-place electronic balance, furnace, IR detector, and microprocessor. Solid samples are weighed in a combustion boat on the balance, and then burned in an oxygen atmosphere, reinforced with a timed jet of oxygen, at 1370°C. The amount of carbon dioxide produced is measured by an infrared analyzer, and reported as the percent carbon in the original sample. Analytical operations and calculations are controlled by the microprocessor. Sample pretreatments are suggested by the manufacturer for analyzing liquid samples and for stripping inorganic carbon so that data refer to organic carbon.

Microprocessor

This Leco instrument is a prime example of how not to use a microprocessor. Controlling analytical operations electronically normally improves precision, but units of measure should be maintained. For this system, the raw data pertaining to the amount of carbon dioxide produced from the test sample is unknown, i.e., no absorbance readings are available for the infrared (IR) detector. The automated but unknown calculations performed on the carbon dioxide content (integrated response) include compensation for the flow rate of the carrier stream (oxygen) and the temperature of the cell of the IR module. Seven sections of the IR calibration curve have been linearized, but the chosen sections cannot be correlated to carbon dioxide levels. The daily response of the system is compared to a number (16384) with no units. Programming is hard wired and proprietary. The calibration procedure is fixed, and the operator cannot adjust - merely accept or reject.

Balance

The CR-12 is provided with a 3-decimal place, top-loader, electronic balance that is connected to the microprocessor. For sample weights between 200 1000 mg, it is accurate within one percent. For weights between 20 and 100 mg, the accuracy is only about 4%. Below 10 mg, the microprocessor breaks down and produces erroneous data. As it is a top loader balance, it is very susceptible to drafts and vibrations. Fortunately weights can be entered into the microprocessor manually, and thus the balance is not used. Unfortunately, the microprocessor has been programmed so that it will only accept weights to 3-decimal places.

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Samples are placed in combustion boats which proved reusable and capable of holding aqueous samples for extended periods of time, eg., long enough to permit analyses or drying of sample. These boats are an improvement over those used for the Leco WR-12; the latter are so porous that aqueous solutions cannot be handled, and are not reusable.

Furnace

Samples are burned in a furnace at a programmable temperature with 1370°C being recommended. Oxygen flow is maintained through the furnace, and a jet of oxygen can be directed at the sample for a programmable period of time. This module is the heart of the Leco instrument and superior, in the author's opinion, to any other on the market.

Infra-Red Detector

Without units of measure, it could not be evaluated.

Although the Leco CR-12 was designed primarily for analyzing solid material for carbon, it is recommended for general application to the environmental field. As such, the preliminary experimental phase concerned testing its flexibility with the hope that the instrument could be used to determine total organic carbon.

Aqueous Samples

The Leco manual described a procedure for determining the carbon content of liquid samples. If feasible, the method would permit the determination of total carbon and assure that volatile carbon constituents were included in the final result. Aqueous samples were weighed and then blanketed with magnesium oxide; the mixture was then analyzed by the normal procedure for solid samples. Magnesium oxide took up the water and controlled the burning rate. When tested, however, the moisture traps within the instrument became clogged after only two or three samples had been analyzed.

Dried Samples

The simplest way of preparing aqueous samples for the CR-12 is to dry them at 103°C. The problem is correlating the carbon data to the original sample. Results for chemical standards showed that the drying procedure resulted in small losses of organic carbon and large losses of soluble inorganic carbon. Moreover, these losses depended upon the chemical composition of the aqueous phase. Limited data suggested that the composition of the dried residue was affected by pH changes during the drying step. To compensate, test solutions were adjusted to pH 3 prior to drying; analyses of these residues showed excessive losses of soluble organic components. Other drying techniques have not been investigated yet.

As an acceptable drying technique was not available for the whole sample, the use of the CR-12 had to be restricted to particulates. By filtering the sample and analyzing the dried particulates, problems arising from loss of volatile organics and soluble inorganic compounds were minimized. If the insoluble inorganic carbon forms could be removed by pretreatment, a test for particulate organic carbon could be developed.

Sewage samples were composted, and the dried particulates analyzed for carbon after: no pretreatment, pretreatment, and pretreatment in the presence of a calcium carbonate spike. In the absence of spikes, pretreatment reduced the carbon content of the particulate fraction by 2 to 4%, and this loss was considered acceptable. In the first series of tests, the wet residue on the filter paper was washed with either 0.1 N hydrochloric acid or 0.1 N sulphuric acid. This treatment only removed 15% of the carbon from the calcium carbonate spike. A more rigorous treatment was then tried. Aqueous samples were treated with either 0.1 N hydrochloric or 0.1 N sulphuric acid, and nitrogen gas was bubbled through the mixtures; the samples were then filtered, and the dried residues analyzed for carbon. This second treatment only removed 23% of the carbon in the calcium carbonate spikes. All tests were conducted in duplicate or triplicate, and the precision was very satisfactory in that the spread of carbon values was less than 0.5% C. Hydrochloric acid discoloured the moisture traps in the CR-12. In view of these results, development of a particulate organic carbon test was stopped.

Particulate Total Carbon

Previous study³ had shown that the Leco WR-12 instrument could be used for the determination of carbon in the particulate fraction of water samples. The final step of the current study was to adapt this method to the CR-12 model. Details of the proposed procedure are given in the appendix, and supporting data is described in Section III of this report. Basically, the proposed procedure entails filtering an aliquot of

well-mixed sample (homogenized if necessary) through a glass fibre filter paper (Reeve Angel 934AH-4.25 cm diameter). After drying at 103°C overnight, the filter paper plus residue are analyzed for carbon using the Leco CR-12 carbon system.

Calibration - General

To calibrate the Leco CR-12 system, a standard of known composition is weighed and analyzed in triplicate. The microprocessor produces a calibration constant, and there is no zero or blank adjustment. The operator can set conditions pertaining to the timing of the burn and collection of data.

Leco recommends that the carbon content of the calibration standards be similar to that expected in test samples. For the selected calibration standard, the carbon content must not exceed 65.5% and the weight analyzed should reflect its carbon content. In effect Leco recommends that the system be calibrated with 50 to 150 mg of carbon. For the proposed system potassium biphthalate (47.05%) was selected as the calibration standard, and two calibration points were evaluated: 50 mg C and 5 mg C.

For calibration, the standard was weighed on a 5-decimal place Mettler balance, and manually entered into the microprocessor module. However, only three decimal points could be entered, and thus the technician was obliged to select a sample weight such that the effect of the fourth decimal place was minimal. The Leco microprocessor program was designed for the top-loader balance (3-decimal places), and cannot accept more accurate weighings.

Before calibration, two or three samples should be run to condition the system. Calibration itself requires triplicate analyses of the standard. If the operator does not like the quality of any burn, the entire process must be repeated, i.e., no deletions or additions. The microprocessor evaluates the calibration data in terms of carbon dioxide production, the flow rate of the carrier gas (oxygen) and the temperature of the IR cell. After calibration the microprocessor prints out a calibration factor which remains in force until the channel

to 1.02 (average 0.96) depending upon the chemical nature of the calibration standard (barium carbonate - 6% C to potassium biphthalate - 47% C), the quantity of standard selected for calibration (10 to 100 mg), and the preselected experimental conditions (10 fold changes). For constant calibration conditions, the factor averaged 0.960 ($\sigma = 0.034$ where $n = 13$). Thus between run calibration control was acceptable.

The Leco CR-12 system has no blank control setting, but does not appear to need one. Whenever combustion boats were analyzed, as samples, their carbon content was negligible. The average carbon content of combustion boats plus glass fibre filter papers was 0.0085% ($\sigma = 0.019\%$ where $n = 22$). Calibration Point = 50 mg C.

The Leco CR-12 was calibrated using 100 mg potassium biphthalate (50 mg C) and the linearity of the calibration was checked by analyzing different weights of the same standard. The actual amount of carbon in the samples ranged from 2 to 118 mg. To evade the three decimal point limitation on sample weight, 0.100 g was entered manually as the weight for all test samples, and the resultant carbon values corrected. (For calibration the actual weight of sample must be fed to the microprocessor, but an arbitrary weight is sufficient for samples; the microprocessor merely calculates the % carbon for whatever sample weight the operator enters.) As can be seen from the data in Table 1, carbon recoveries increased from 81 to 102% as the amount of sample increased from 2 to 118 mg C. However, the linearity of the pre-processed calibration curve was acceptable for the range 9 to 118 mg C. To further check the system, 100 mg asparagine (32 mg C) and 100 mg calcium carbonate (12 mg C) were analyzed; recoveries were 99 and 98% respectively. It appeared that calibrations based on 100 mg potassium biphthalate could serve for the analysis of samples provided their suspended solids contained 10 to 100 mg C. This condition imposed a severe limitation on the value of the instrument. For some samples, volumes in excess of one litre would be required while other samples would clog the filter before this necessary weight of particulates was accumulated.

Although not recommended by Leco, calibration with a smaller amount of carbon had to be considered. Thus the system was calibrated with 10 mg potassium biphthalate (5 mg C). Low weights proved awkward as the microprocessor would only accept weights greater than 10 mg and only record them to the nearest mg. By using the 5-decimal place balance and carefully selecting the number of crystals, the effect of the fourth decimal place on the overall sample weight was minimized. Once calibrated in this fashion, various weights of standards were analyzed by entering 100 mg, and correcting the carbon data produced by the microprocessor. The procedure was conducted three times, and the results for potassium biphthalate, asparagine, and calcium carbonate are included in Table 1 and illustrated in Figure 1. As the carbon content of the test samples increased from 1.4 to 47 mg, the percent recoveries increased slightly but the bias was considered acceptable. The average carbon recovery was 99.3% ($\sigma = 2.8\%$ where $n = 20$). Figure 1 confirmed that blank problems were minimal.

By using two calibration points, the system could handle the expected range of particulate carbon in Water Quality samples. The Leco system provides four calibration channels, and thus the appropriate channel could be selected for a given sample.

Quality Control (QC)

As the calibration of the IR detector cannot be adjusted, QC is restricted to monitoring the calibration factor and recovery checks.

For each run, the channel, calibration point, and calibration factor are recorded. For the high level (50 mg C), QC-A is 100 mg asparagine and QC-B is 100 mg calcium carbonate. For the low level (5 mg C), QC-A is 10 mg asparagine and QC-B is 10 mg calcium carbonate. These QC compounds check recovery of both organic and inorganic forms of carbon. Moreover, their carbon contents are appropriate because the expected carbon in

Precision

Recovery and precision of standards were determined at various carbon levels for both calibration points (Table 2). When the calibration point was 50 mg C and the carbon content in the samples exceeded 25 mg, recoveries were accurate within 0.2% carbon and precision, as measured by standard deviation, ranged from 0.4 to 0.8% C. When the calibration point was 5 mg C and the carbon content in the samples ranged from 1.0 to 15 mg C, recoveries were accurate within 0.8% C, and precision ranged from 0.5 to 1.2% C.

Multiple analyses were conducted on composted sewage samples (Table 3). The results showed that precision, as measured by standard deviation, ranged from 0.28 to 9.4 mg l⁻¹ C as the suspended solids increased from 28 to 3000 mg l⁻¹. This level of performance was quite acceptable.

Precision data for a dried digested sludge were included in Table 3; the relative standard deviations for total solids and carbon were 1.36% and 1.56% respectively. For these replicates, 2 ml of sludge were pipetted directly into the combustion boats; the mixtures were dried overnight at 103°C, and then analyzed for carbon. The results were surprisingly precise, but the data could not be correlated to specific forms of carbon. If, however, a suitable sample pretreatment can be developed, the Leco CR12 is apparently capable of producing precise data for these problem samples.

Recovery Data

To evaluate the accuracy of the CR12, organic compounds with carbon contents ranging from 20 to 77% were analyzed (Table 4). Although most of the test compounds were analytical grade, they were collected from open shelves throughout the building, and the quality varied at the time of testing. For the first set of

bipthalate), and approximately 100 mg of each compound was analyzed; the Leco 3-decimal place balance was used to weigh test compounds but not for calibration. The average recovery of carbon from these compounds was 100.0% ($\sigma = 2.34\%$ C where $n = 39$). For the second calibration point (5 mg C), approximately 10 mg of each test compound was analyzed; all weighings were conducted with the 5-decimal place Mettler balance. Again, the average carbon recovery was 99.5% ($\sigma = 2.08\%$ C where $n = 41$). These results indicated that the Leco CR-12 system was suitable for a wide variety of organic chemicals and presumably accurate for Water Quality's samples.

Intercomparison

No intercomparison was conducted as the Water Quality Section does not have an alternate system for determining carbon in samples containing particulate matter.

CONCLUSIONS

- 1) The Leco CR-12 system determined the carbon content of particulates with acceptable levels of accuracy and precision.
- 2) The limitations imposed on the analyst by the manufacturer severely limit the value of this system. If the role of the microprocessor cannot be modified, the instrument must be considered as a stop gap until a better system is available.

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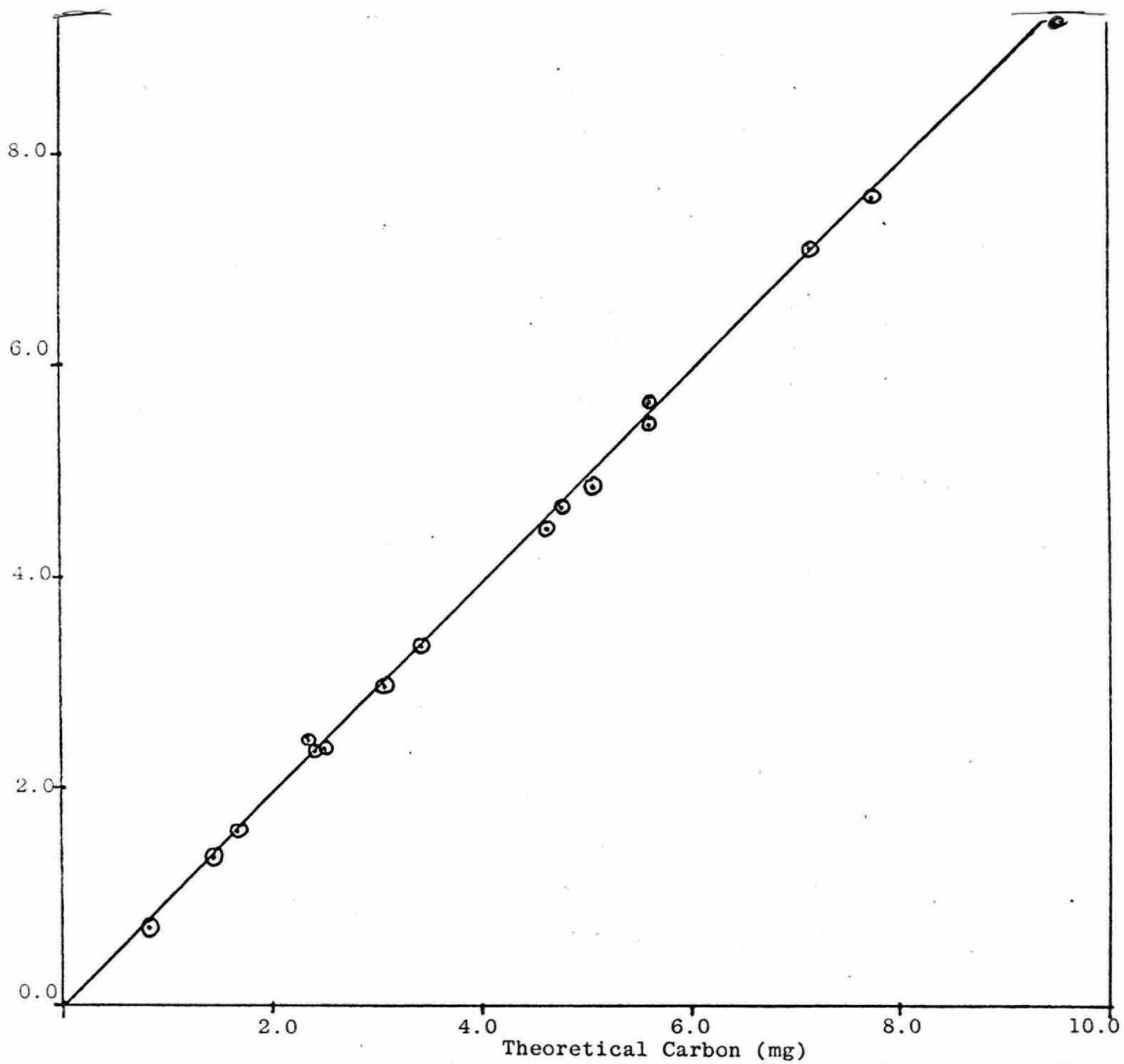


Figure 1, Calibration Check of Leco CR-12 System (Calibration Point: 5 mg C)

Table 1

CALIBRATION DATA

Chemical Standard	Calibrated With 100 mg Potassium Biphthalate			Calibrated With 10.0 mg Potassium Biphthalate		
	Theoretical Carbon (mg)	Measured Carbon (mg)	Recovery (%)	Theoretical Carbon (mg)	Measured Carbon (mg)	Recovery (%)
Potassium biphthalate	1.96	1.59	81			
	2.40	2.02	84	2.36	2.45	104
	3.51	3.26	93	2.40	2.38	99
	4.08	3.43	84	2.48	2.37	96
	4.48	4.29	96	3.42	3.35	98
	4.78	3.99	83	4.66	4.47	96
	5.12	4.89	96	4.77	4.67	98
	5.13	4.54	89	5.59	5.44	97
	9.20	8.75	95	7.16	7.07	99
	9.46	9.19	97	7.75	7.55	97
	9.55	9.18	96	9.50	9.28	98
	9.60	9.26	96			
	13.75	13.61	99	11.10	11.25	101
	14.20	13.70	96	14.45	14.71	102
	15.35	15.04	98			
	15.55	15.26	98			
	17.80	17.07	96			
	18.15	17.86	98			
	18.95	18.43	97	18.80	19.43	103
	20.95	20.57	98			
	23.10	22.79	99			
	23.40	23.06	99			
	23.50	23.08	98	23.90	24.91	104
	25.40	25.24	99			
	35.40	35.18	99			
	46.20	46.29	100			
	47.05	47.00	100	47.05	47.40	101
	50.50	50.6	100			
	50.80	50.8	100			
	59.00	58.65	100			

Table 1 (cont'd)

Chemical Standard	Calibration With 100 mg Potassium Biphthalate			Calibrated With 10.0 mg Potassium Biphthalate		
	Theoretical Carbon (mg)	Measured Carbon (mg)	Recovery (%)	Theoretical Carbon (mg)	Measured Carbon (mg)	Recovery (%)
Potassium biphthalate	70.40	70.81	101			
	82.50	83.4	101			
	94.50	95.81	101			
	99.0	101	102			
	106.0	108.4	102			
	118.0	120.5	102			
Asparagine	32.0	31.7	99	1.68	1.62	96
				3.09	2.98	96
				15.9	16.2	102
Calcium Carbonate	12.0	11.7	98	0.80	0.72	90
				1.42	1.39	98
				5.60	5.65	101

Table 2

ANALYSES OF STANDARDS - RECOVERY AND PRECISION

Calibration Point* (mg C approx.)	Standard Chemical	Sample Size (mg C)	Theor. C (%)	Measured C (%)	No Observations	St'd Dev'n (% C)	Precision Type
50	Potassium biphthalate	125	47.05	47.0	12	0.41	Within Run
		50	47.05	47.0	10	0.58	Within Run
		50	47.05	47.0	10	0.76	Within Run
		25	47.05	47.2	9	0.51	Within Run
		5	47.05	41.7	10	3.35	Within Run
		5	47.05	39.4	4	1.05	Within Run
		3	47.05	44.6	5	1.65	Within Run
5	Asparagine	15	31.97	31.15	3	0.03	Within Run
	Potassium biphthalate	5	47.05	46.93	19	1.18	Between Run
	Asparagine	3	31.97	31.39	19	0.59	Between Run
	Calcium Carbonate	1	11.99	11.76	19	0.51	Between Run

*System was calibrated with potassium biphthalate; weights of standard were approximately 100 and 10 mg for 50 and 5 mg C respectively

Table 3

PRECISION, REPLICATE ANALYSES OF SEWAGE COMPOSITES

Sample Type	Solids				Carbon Analyses		
	Average (mg l ⁻¹)	No. <u>Observations</u>	St'd Dev'n (mg l ⁻¹)	St'd Dev'n for <u>Routine Procedure*</u>	Average (mg l ⁻¹)	No. <u>Observations</u>	St'd Dev'n (mg l ⁻¹)
Final Effluent	27.80	25	1.66	3.97	5.50	25	0.28
Raw Sewage	100.2	24	2.00	4.34	33.20	24	0.84
Mixed Liquor	3150	5	69	64	527	5	4.7
	3230	5	53	64	564	5	9.4
Sludge	22,500	25	306	200	6660	25	104

Solids: Suspended solids were determined on the raw sewage, final effluent, and mixed liquor samples using 4.25 instead of 9.0 cm diameter glass fibre filter paper. Total solids were determined on the sludge using a 2.0 ml aliquot of homogenized sludge instead of 50 ml of sample as received.

*Precision data refers routine test procedures for SIW group

Table 4

RECOVERY OF ORGANIC COMPOUNDS

Organic Compound*		Calibration Point and Sample Wt: 100 mg Approximately			Calibration Point and Sample Wt: 10 mg Approximately	
Type	Name	Carbon (%)	Measured Carbon (%)	ΔC^{**} (%)	Measured Carbon (%)	ΔC^{**} (%)
Acid	Abietic ^y	74.6	77.1	+2.5	72.1	-2.5
	Ascorbic	40.9	42.0	+1.1	40.8	-0.1
	1,4-Dihydroxybenzoic	54.5	50.6	-3.9	49.2	-5.2
	Lauric	72.0	74.8	+2.8	72.0	0
	Malonic	34.6	36.1	+1.5	33.5	-0.9
	Malic	35.8	37.3	+1.5	34.8	-1.0
	Myristic	73.7	75.6	+1.9	75.6	+1.9
	Nicotinic	58.5	--	--	56.7	-1.8
	Palmitic	75	76	+1	76	+1
	Pyrrolidinecarbodithioic	35.0	--	--	36.5	+1.5
	Tannic ^y	53.6	47.2	-6.4	--	--
	Tartaric	32.0	31.8	-0.2	--	--
Aldehyde	Trimethylacetaldehyde sodiumdisulphite ^y	31.6	26.5	-5.1	--	--
Amide	Acetamide ^y	49.3	42.5	-6.8	--	--
	Acetanilide	71.1	72.2	+1.1	71.5	+0.4
	Asparagine	32.0	31.7	-0.3	30.8	-1.2
	Ethylcarbamate	40.4	42.6	+2.2	40.3	-0.1
	Nicotinamide	59.0	60.9	+1.9	60.1	+1.1
	Phenacetin	67.0	65.1	-1.9	69.3	+2.3
	Sulphanilamide	41.8	42.6	+0.8	42.1	+0.3
	Urea	20.0	20.0	0	18.9	-1.1
Amine	Adenine	44.4	44.4	0	43.5	-0.9
	Aminophenazone	65.0	66.2	+1.2	63.9	-1.1
	Cytosine	43.2	40.1	-3.1	38.8	-4.4

Table 4 (continued)

Organic Compound*		Calibration Point and Sample Wt: 100 mg Approximately			Calibration Point and Sample Wt: 10 mg Approximately	
Type	Name	Carbon (%)	Measured Carbon (%)	ΔC^{**} (%)	Measured Carbon (%)	ΔC^{**} (%)
	Guanine	39.7	40.2	+0.5	43.9	+4.6
	Hydroxyquinoline	74.7	--	--	73.7	-1.0
	N-1-naphthylethylenediamine dihydrochloride	55.6	56.0	+0.4	55.1	-0.5
	Nitroaniline	52.1	53.7	+1.6	52.1	0
	σ Phenylenediamine ^y	66.6	67.8	+1.2	69.4	+2.8
	m-Phenylenediamine dihydrochloride	39.8	39.9	+0.1	40.7	+0.9
	Thymine	47.6	49.5	+1.9	46.8	-0.8
	Uracil	42.8	44.0	+1.2	41.2	-1.6
Amino Acid	Aspartic acid	36.1	--	--	33.2	-2.9
	Glutamic acid	43.6	43.1	-0.5	40.7	-2.9
	Lysine monohydrochloride	39.4	36.4	-3.0	40.6	+1.2
Quaternary	Aminoethyltrimethylammonium chloridehydrochloride	34.3	34.8	+0.5	36.2	+1.9
	Tetramethylammonium chloride	43.8	43.9	+0.1	41.1	-2.7
Ester	Ethyl stearate	77	78	+1	79	+2
	Potassiumhydrogen phthalate	47.1	47.2	+0.1	46.8	-0.3
	Sodiumhydrogentartrate	25.3	--	--	25.0	-0.3
Organic P	Phosphoserine	19.5	18.9	-0.6	15.6	-3.9
Sugar	Glucose	39.9	41.9	+2.0	37.9	-2.0
	Sucrose	42.1	--	--	42.6	+0.5
Aromatic Benzene	Dinitrobenzene	42.9	44.0	+1.1	41.2	-1.7
	S-Benzylthiuronium chloride ^y	47.4	49.7	+2.3	46.4	-1.0

Table 4 (continued)

Notes: (1) The carbon content of the test samples ranged from 20 to 77%, and averaged 47%

(2) When calibrated and operated at the 100 mg level using the 3-decimal place Leco balance, the average recovery of the organic compounds was 100.04% ($\sigma = 2.34\%$ where $n = 39$).

(3) When calibrated and operated at the 10 mg weight level via a 5-decimal place Mettler balance, the average recovery of the organic compounds was 99.52 ($\sigma = 2.08\%$ where $n = 41$).

* Test compounds were obtained from opened bottles, and had been stored for six months to five years.

** ΔC is the difference between expected and measured recovery

y Compounds were technical grade and/or appeared to have changed composition.

APPENDIX

Procedure For The Determination of Total Carbon In Particulate Matter

3.1 Leco CR-12 Carbon System consisting of:

- 1 780-000 Control Console
- 1 781-700 Measurement Unit
- 528-203 Sample boats
- 1 501-062 Boat puller assembly

- 1 oxygen cylinder regulator
- 2 metal cooling trays
- 1 micro spatula
- glass wool

3.2 Millipore filter apparatus

3.3 Reeve Angel glass fibre filters 934AH (4.25 cm diam.)

3.4 Filter racks

3.5 Mettler 5-place balance # HL52

3.6 1 5 ml graduated wide tip pipette

3.7 1 50 ml volumetric wide tip pipette

3.8 1 100 ml volumetric wide tip pipette

3.9 1 250 ml measuring cylinder

4. Reagents

4.1 Potassium Hydrogen Phthalate, $\text{HOCOC}_6\text{H}_4\text{COOK}$, analytical grade, crushed and dried

4.2 L-Asparagine, $\text{NH}_2\text{CO}.\text{CH}_2\text{CH}(\text{NH}_2).\text{COOH}.\text{H}_2\text{O}$, analytical grade

4.3 Calcium carbonate, CaCO_3 , analytical grade, powder form

4.4 Magnesium Perchlorate $\text{Mg}(\text{ClO}_4)_2$, anhydrous crystals

4.5 Oxygen gas, dry, cylinder size K stores #52-C-186

5. Procedure

5.1 Sample preparation

Final effluents, mixed liquors, raw sewages and industrial wastes must be filtered through a millipore filter apparatus using glass fibre filter papers, and dried overnight at 103°C . Generally, a 200 ml aliquot

raw sewage. The filtrate is sent to the river lab for dissolved organic and dissolved inorganic carbon analyses.

- 5.2 Refer to the Leco CR-12 manual before following the instructions described below:
- 5.3 Check the anhydron traps on the front of the measurement unit. They should be clean and dry, and the anhydron should not be caked. If a trap has to be replaced, pack it firmly with large pieces of glass wool, twisting the wool as it goes through the tube. The trap nearest the furnace should be packed with glass wool until it is half filled and then filled to about $\frac{1}{2}$ " from the top with anhydron. The metal screen should be placed in the bottom of the tube. The trap nearest the console should have glass wool at both ends with about 3" anhydron between and a screen on top. Lightly grease the O rings on the trap connectors before replacing traps.
- 5.4 Open the oxygen regulator all the way and set the pressure on the gauge to 30 p.s.i.
- 5.5 Press 'reset' and 'system update' on the control console. Step through the functions offered by pressing 'no' until 'set furnace temp.' appears on the display. Change the furnace temperature from 1000°C to 1370°C using the numerical keys on the console. The digit which flashes is the one which will be changed.
- 5.6 Press 'system update' again and go to 'display constants'. Check that the minimum acquisition time is 60 seconds. Lance delay 50 seconds. Comparator level 2 and answer precision 5. (The clean counter and clean interval are not used. Answer precision constants should not be altered manually.)

.1g KHP into it. Press 'enter' when weight is satisfactory and stable. Press analyse.

- 5.8 When the furnace light glows, push the sample boat to the back of the furnace using the boat puller.
- 5.9 Upon completion of analysis the console will print out the measured % C of the sample. Repeat analysis. The first two samples are run to condition the instrument, and their results are not to be used. Allow boats to cool on the plate by the furnace before removing to cooling tray.

5.10 Calibration:

- 5.10.1 Weigh .010 - .015g KHP into 3 sample boats on the five-place balance and record weights. (Instrument will not accept a sample weight of less than .010g). *enter below*
- 5.10.2 Press reset and system update 'in order to access calibrate burns' on the display. Press 'yes'.
- 5.10.3 The display will ask for a calibration number. '3' has been arbitrarily selected for calibrating with .01g KHP.
- 5.10.4 Carbon standard % will be 47.05 for KHP. Press 'enter' if this is displayed, or enter manually.
- 5.10.5 The console will now display 'enter sample weight'. Press 'manual' and enter the sample weight correct to 3 decimal places. There is no need to press 'analyse' when in the calibration mode.
- 5.10.6 When the furnace is ready, the indicator light will glow and the first standard may be inserted. After each standard has been analyzed, the console will print out the measured % C and oxygen flow during analysis. When calibration is complete, the new

strument will return to its operate mode. Record the values for the standards and oxygen flow and calibration factor on the control data sheet. Oxygen flows should be between 3.250 and 3.280 l/min.

- 5.11.7 Weigh accurately on the 5-place balance .010-.015 g Asparagine and .010-.015 g Calcium Carbonate and run as QCA and QCB respectively. Enter 0.100 g weight manually and calculate result as follows:

$$\% C = \frac{0.1g}{\text{actual weight}} \times \% C \text{ reading}$$

Values must be within 32% ± 1 and 12% ± 1 before proceeding to analyse samples. Record values on control data sheet.

- 5.12.8 Samples may now be run by entering manually 0.100 g as sample weight. Press the filter papers into the boats so that they line the bottom and side and do not overlap the edges of the boats.

- 5.13.9 Use .010 - .015 g KHP as an in run standard every 10 samples. *Calc. expected % and compare with observed.*

- 5.14.10 The anhydron trap nearest the furnace should be changed whenever it becomes moist, caked or covered with carbon particles.

6.0 Calculation and Reporting

$$\text{mg/l PTC} = \frac{\% C \text{ Reading} \times 1000}{\text{Aliquot (ml)}}$$

Results are reported to three significant figures.

7. Instrument Shutdown

- 7.1 Press 'system update' and step through functions to set furnace temperature. Change the temperature from 1370°C - 1000°C by typing out 1000 on the keyboard. The furnace should not be allowed to go below 1000°C except for non-routine maintenance.



(7274)

MOE/DET/ALPF

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Crowther, Joan

Determination of

particulate carbon : alpf

c.1 a aa